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TECHNICAL REPORT PBA-TR-QAL-91-1

MULTIAGENT MINICAMS PERFORMANCE & DEPENDABILITY EVALUATION

by

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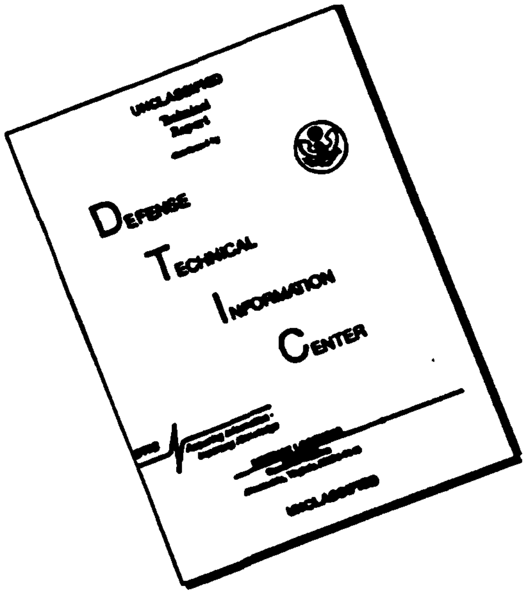

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Two multiagent MINICAMS were challenged in a laboratory setting with HD, GB, and VX at concentrations equivalent to sampling air at 0.2, 0.5, 1.0 and 2.0 times TWA for each agent. Both instruments demonstrated LOQ quantities well below 1 TWA equivalent for all three agents. The MINICAMS were then installed in a mobile laboratory and field tested by injecting GB standards into a 75 foot heated sampling line inside a storage igloo. Sixty four sets of data were collected on each instrument over an 8 day period in which the mobile lab was intermittently driven over rough roads. Statistical analysis of the resultant data indicates both instruments will detect GB with a limit of quantitation (LOQ) less than 0.35 times TWA but with a small interval between TAL and LOQ indicating a high risk of false negatives at 1 TWA.

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I. Introduction.

Monitoring of chemical storage depots incorporates a combination of bubblers, blue band detector tubes, and M8/M8A1 automatic chemical detectors. These methods suffer from expense, time, sensitivity and selectivity restraints. A potential solution to upgrade current monitoring procedures is to use Miniature Automatic Continuous Air Monitor Systems (MINICAMS) mounted in a mobile vehicle with heated sample lines that would be inserted into a chemical munition storage igloo. The advantages of MINICAMS are expected to be cost savings, sensitivity improvement, and shortening of analysis times.

Pine Bluff Arsenal was tasked with performing field trials on the MINICAMS to determine the reliability and functional capabilities of the units. Two multiagent MINICAMS were purchased from CMS Research Corp. by the Detection Directorate, Chemical Research Development and Engineering Center (CRDEC), Aberdeen Proving Ground, and loaned to the Pine Bluff Arsenal (PBA) for testing and evaluation. They were installed in a vehicle specially modified to support a mobile laboratory operation. The following report documents the laboratory and field functioning tests which were performed.

II. Experimental.

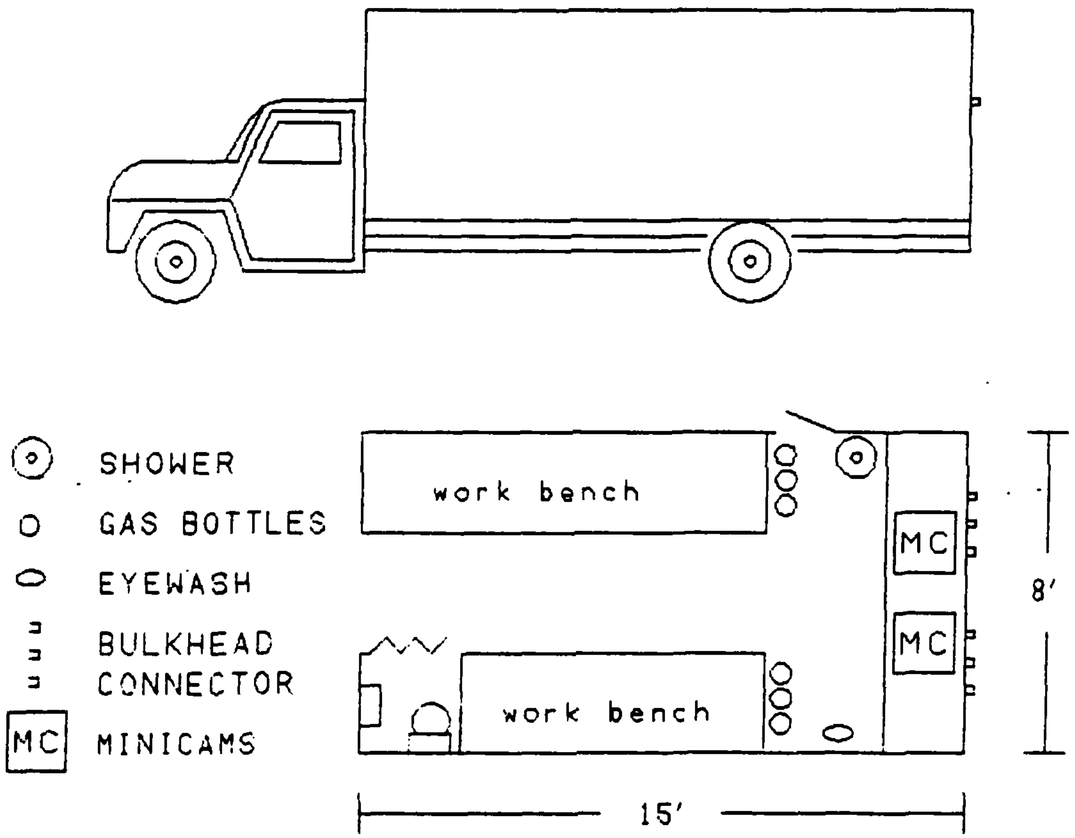
A. Instrumentation.

The multiagent MINICAMS S/N 1394 and 1395 air monitoring and alarm systems were purchased from CMS Research Corp., Birmingham, Alabama. The systems consist of sorbent tube collection devices, capillary GC columns and flame-photometric detectors with automated timing circuits and alarms (See Diagram 2, page 10). The entire system is contained in a single chassis which includes the sampling and analytical system, the controller, and the display. Basic instrument operating parameters (gas flows, detector temperature, etc.) can be set by the operator. Concentration readings and alarm conditions are indicated on the liquid crystal display (LCD) and can be transmitted to a dual pen recorder, a printer, a floppy disk drive, and a remote computer. The recorders and floppy disc drives were purchased from CMS Research Corp.

A two ton dual rear wheel Utilimaster Ford truck was modified into a mobile laboratory and housed all necessary equipment. See Diagram 1, page 8, for the general layout.

The MINICAMS, when in the mobile laboratory, were powered by 2 parallel 2000 watt TRIPP Lite inverters from Trippe Manufacturing Co. The inverters supplied uninterruptable 120 vac. from a 12 volt battery pack, which in turn was charged by an on-board 120 vac. Onan generator. The TRIPP inverter was modified from a square wave to a sine wave output because it became evident that the MINICAMS circuitry would not operate on a square wave. The model 1531-107B-G288X vacuum pumps from Gast Manufacturing Co. for air sampling were powered directly from the Onan generator. The heated sample

lines purchased from CMS Research Corp. were powered independently of the van from a 120 volt power line run to the igloo. The 75 foot sample lines were 1/4" o.d. teflon wrapped with heat tape for uniform heating. The operating temperature was approximately 60 degrees C. The sample lines were connected to the outside of the truck with stainless steel Quick-Connect fittings.

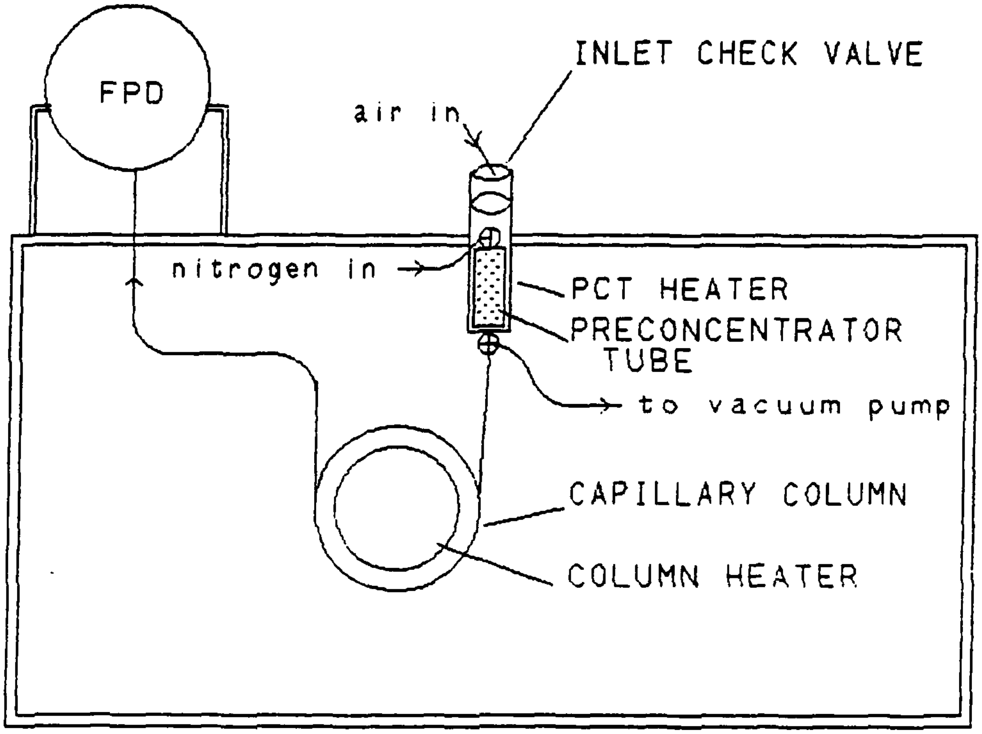


MOBILE LABORATORY FLOOR PLAN Diagram 1

B. Principles of Operation.

The MINICAMS were set to sample air through an inlet check valve and preconcentrator tube (PCT) for 180 seconds using a vacuum pump. The vacuum pump flow rate was set at 1 liter/minute. After sampling 3 liters of air a valve switched from vacuum to nitrogen (20 psi. head pressure), and directed it through the PCT into a DB-1 equivalent capillary column (0.32 mm. i.d., 15 meters, coated with a 4 micron methyl silicone film). The PCT was ballistically heated to 220 degrees C. for 30 seconds to purge the agent which collected on the capillary column. The capillary column was then ramped from 30 to 180 degrees C. in 45 seconds to separate the agent from interferents and elute it into a flame photometric detector (FPD). The FPD is especially sensitive and selective to phosphorus and sulfur containing compounds (See Diagram 2, page 10). A choice of optical filters in the photomultiplier tube brackets either 526 nanometers for phosphorus or 394 nanometers for sulfur. Agents were identified by retention time and quantitated by peak height. Agent concentrations were automatically calculated into TWA equivalents based on the standard used to calibrate the instrument and displayed on the LCD. ᴬ instrument alarm and warning light were configured to alert the operator of high concentrations of agent. Data from the test program was assessed by "CERTIFY" software used in the Laboratory Quality Assurance Program at Pine Bluff Arsenal.

A dual pen recorder was used to help distinguish the agent from possible interferences in the chromatogram. A floppy disc was used as an optional means of data collection. Data was primarily collected by writing LCD displayed results on data sheets.



MINICAMS FLOW DIAGRAM

Diagram 2

C. Acceptance Requirements.

All agents were injected into each instrument according to the scheme shown in Table 1. TWA challenges were calculated by assuming 100% trapping efficiency when sampling air at TWA agent levels. Agent samples were then diluted so that 5 ul contained the amounts expected when sampling air at 1 TWA.

TABLE 1

Acceptance Testing Scheme for MINICAMS for all Agents

Injection Scheme						Acceptance (TWA) Range
one	0	ul	injection	= 0	TWA	--- N/A ---
one	1	ul	injection	= 0.2	TWA	0.15---[0.20]---0.25
five	2.5	ul	injections	= 0.5	TWA	0.38---[0.50]---0.62
ten	**5**	**ul**	**injections**	**= 1**	**TWA**	**0.75---[1.00]---1.25**
five	10	ul	injections	= 2	TWA	1.50---[2.00]---2.50

This scheme calls for making 22 injections for each agent on each instrument.

For the MINICAMS to perform acceptably, 95% of the 5.0 ul or 1 TWA equivalent values must be in the range of 0.75 to 1.25 TWA. Additionally, 75% of the remaining values must be within ± 25% of their theoretical values, except for the 0.2 TWA equivalent values where the results are desired but not required (See Appendix 1).

Field Functional testing was carried out using the scheme in Table 2. One set consisted of 4 data points as shown. Each instrument was challenged with 64 sets under varying field conditions.

TABLE 2

Data Sets Collected for Field Functional Testing

Injection Scheme				Target (TWA) Range
5	**ul = 1**	**TWA = 0.3**	**ng GB**	**0.75---[1.00]---1.25**
2.5	ul = 0.5	TWA = 0.15	ng GB	0.38---[0.50]---0.62
10	ul = 2	TWA = 0.6	ng GB	1.50---[2.00]---2.50
0	ul = 0	TWA = blank		--- N/A ----

Injections were made in the order shown.

D. <u>Agents Used in Testing</u>.

Neat SARM agents were obtained from CRDEC, Aberdeen, Maryland. The GB was 95.7 mole% pure by NMR; the HD was 97.6 mole% pure; and the VX was 95.9 mole% by NMR. Dilutions were made into hexane. All stocks and standards were stored at 0 to -10°C.

E. <u>Test Procedures</u>.

1. <u>General</u>.

The MINICAMS were turned on and gas cylinders opened, with nitrogen and air head pressures adjusted to 20 and 35 psi., respectively. The hydrogen pressure was set to 10 psi. until the flame photometric detector (FPD) block reached approx. 100 degrees C. The multiagent units have an autoignite feature. After the flame autoignited, the hydrogen pressure was increased to 35 psi. The proper optical filter and preconcentrator tube were verified.

The agent to be analyzed and correct input operating parameters were selected on the analog screen. The PARAM push-button and then INCR or DECR push-buttons were depressed to set the sample flow (SAMF) to 100 ml/min (if no linear mass flowmeter (LMF) is available, set SAMF to zero otherwise an error code will be activated during the sampling period). After turning on the vacuum pump, the signal screen was selected by pressing the INCR and PAGE push-buttons. When the MINICAMS unit was in the sampling period (the clock is also visible on this screen), the vacuum pump needle valve was adjusted to obtain 1000 ml/min.

The recorder cables were connected from the recorder to the MINICAMS, and the recorder was zeroed on each channel. The INCR and PAGE push-buttons were used to select the Recorder 1 (R1) screen, and the PARAM and ENTER push-buttons to select the chromatrogram (CHROM) parameter (black pen). The PAGE push-button was then pressed until the Recorder 2 (R2) screen was seen and the above steps were repeated to select the concentration (CONC) parameter (red pen). The chart speed was set to 2 cm/min with both channels set on the 10V range scale.

The working standard was removed from the freezer and stabilized at room temperature. A succession of agent injections was made at the inlet valve and with the aid of the recorder the best agent gate setting for the analyte was determined. A 1 TWA injection was then made and while viewing the main screen the PARAM push-button was pressed until CAL was blinking. When the concentration was displayed, ENTER was immediately pressed to calibrate the instrument at 1 TWA (if the audio alarm has been activated, pressing the ENTER push-button will simultaneously silence the alarm). Several other 1 TWA injections were made to verify that the displayed concentrations remained close to 1.0 TWA. Table 3 shows the operating parameters used for all agents.

TABLE 3

General MINICAMS Operating Parameters

TEMPERATURES °C:	Ambient	(AMB)	:	35 ± 15	
	Inlet	(INL)	:	35 ± 15	
	FPD block	(FPDT)	:	150 ± 15	
	FPD flame	(FLAT)	:	225 ± 50	
	Column, low	(LCOL)	:	30 ± 15	
	Column, high	(HCOL)	:	180 ± 15	
	PCT heater, low	(LPCT)	:	35 ± 30	
	PCT heater, high	(HPCT)	:	220 ± 5	
PRESSURES psi:	Air	(AIPP)	:	35 ± 5	
	Nitrogen	(N2PR)	:	20 ± 5	
	Hydrogen	(H2PR)	:	35 ± 5	
SAMPLE ml/min:	Flow rate	(SAMF)	:	1000 ± 20	
VOLTAGE v.d.c.:	FPD photomultiplier		:	1400 ± 50	
TIMES sec.:	Purge 0 - 120	Sample		120 - 300	
	Desorb 0 - 30	Column		30 - 75	
	Inject 120 - 130	Inlet		0 - 300	
	FPD zero 5 - 15	Agent Gate		~47 - 57	GB
				~40 - 50	VX
				~70 - 80	HD
PHOTOMULTIPLIER OPTICAL FILTER :	Yellowish-green	for		Agents GB and VX	
	Purple	for		Agent HD	
PRECONCENTRATOR TUBE :	Hayesep D	for		Agents GB and VX	
	Tenax GC	for		Agent HD	

V-G conversion pads were installed above the inlet check valve for VX analyses. Injections for VX were made by depositing agent directly onto the V-G pad.

2. <u>Acceptance Testing</u>.

Acceptance testing was performed in accordance with a plan submitted by the Test and Evaluation Office: Research, Development & Engineering Support Directorate; CRDEC (Appendix 1). The instruments were first installed in the Product Assurance Lab at the Pine Bluff Arsenal. Both instruments S/N 1394 and 1395 were calibrated by injecting 5 ul of working standard into the inlet check valves. Initially, CMS Research Corp. recommended using calibration concentrations of 0.16 ppm GB, 4.8 ppm HD and 0.016 ppm VX. Injections of 5 ul of these standards are equivalent to sampling air with TWA levels of agent for 8 minutes using the MINICAMS on a 10 minute cycling program. To save time, however, Dan Coleman from CMS Research suggested using a 5 minute cycling program. Because background interferences were occasionally found to occur in the chromatograms when sampling for longer times, additional calibration sets and series of injections were performed on each instrument using GB at 0.055 ppm and HD at 1.62 ppm. These concentrations represent the amounts that would be found in air at a 900 ml/min flow on a 5 minute cycling program with a 3 minute sampling time consistant with the parameters we used while evaluating these instruments (See Table 3). VX was also evaluated using a 0.023 ppm standard on a 15 minute cycling program. A fresh standard was used daily, and in all cases a 10 ul Hamilton syringe was used for injections at 120 seconds into each cycle (See Table 3). After the initial calibration, the instruments were only recalibrated if 2 or more consecutive data points were out of the acceptance range (Table 1). Two series of injections were made for each agent on each unit with each calibration concentration evaluated.

3. Functional Field Testing.

Tests were performed in accordance with the test plan submitted by CRDEC (Appendix 1). The MINICAMS were powered up daily with the mobile laboratory at the laboratory (Bldg. 34-111); the igloo was key obtained; the truck driven to the test igloo site (Bldg. 61-460); and the 75 foot heated sample lines were connected to the external sampling ports. Short pieces of unheated teflon line were used to connect the internal sampling ports to the MINICAMS inlet check valves.

Five microliters of a 0.06 ppm GB working standard were injected directly into the inlet check valves to calibrate the instruments. The operating parameters are listed in Table 3. A fresh standard was used daily and in all cases a 10 ul Hamilton syringe was used for injections. After the initial calibration the units were recalibrated in the field when readings began to fall outside ± 25% of the target range for each concentration. A data set consists of the four points shown in Table 3.

Data set injections were made inside the igloo at the end of 75 foot heated sample lines at 120 to 130 seconds into the MINICAMS cycle. At the 1 l/min. flow rate used, the sample should reach the instrument in 45 seconds, well within the 3 minute sampling time of the cycle. Because no linear mass flowmeters (LMF) were available for these multiagent instruments, the vacuum pump sample flow rates were set at 1000 ml/min using a LMF that was connected to a single-agent MINICAMS. (A single-agent MINICAMS unit was available in the mobile lab but was not a part of the Field Functional Testing.) The vacuum pumps were then switched back to the multiagent units.

Periodically the preconcentrator tubes (Hayesep D) required changing. They were generally changed every few days when the blank began to drift upward following the 10 ul injection of a data set.

After a data set for each instrument was collected at the igloo, the igloo was secured, sample lines were disconnected, and the van driven on paved and gravel roads for about 30 minutes. The MINICAMS and heated sample lines were operated continuously after daily start-up until the end of the daily test routine. Data sets were collected for each instrument over a period of 8 weeks.

Note was made of all instrument error messages and malfunctions on Failure/Maintenance Data Sheets. Agent challenge responses were recorded on Field Functional Test Data Sheets.

At the conclusion of the day, the MINICAMS were turned off and the gas supplies were turned off at the tanks. The igloo was secured, heated sample lines disconnected from the van, electrical power to them disconnected, and the igloo key turned in.

III. Results.

A. Acceptance Testing.

MINICAMS serial number 1394 was subjected to the acceptance testing criteria for chemical agent GB at two different agent standard concentrations: 0.16 ng/ul and 0.055 ng/ul respectively. A total of 44 data points were collected at the 0.16 ng/ul concentration. Of these, 40 were subjected to statistical evaluation using the CERTIFY software provided by the Chemical Stockpile Disposal Program. The 4 points not included were the two blanks that had readings of 0 TWA, and the two injections made at 0.2 TWA that gave readings of 0.20 TWA and 0.18 TWA. Having only two readings at each of these spiking levels made it impossible to include these determinations in the statistical treatment of the data because of inability of the CERTIFY program to accept less than 3 data points at each challenge level. No repeat injections had to be performed for this trial and no outliers were detected. A brief summary of the statistical results for this instrument can be found in Table 4. Table 7 shows the alarm settings generated by the CERTIFY program from the data collected on instrument 1394 using the 0.055 ng/ul calibration standard. Diagram 3 shows the Target vs. Found relationship, % uncertainty, and additional statistical data for acceptance testing of this instrument using the same calibration standard. From Tables 4 and 7, it can be seen that the instrument alarm setting of 0.80 TWA as recommended by the manufacturer is supported by this data. The concentration interval between the LOQ and the TAL is large enough to minimize the risk of obtaining false positives and negatives. Diagram 3 also shows that the uncertainty (with 95% confidence) in the air sample found concentration is $\pm 13.7\%$, which is well within the $\pm$ 25%

guideline set forth in AMC 385-131. For the trials conducted using the GB standard concentration of 0.055 ng/ul on instrument 1394 the results seem to be biased toward higher found concentrations. This indicates that a problem may have occurred during the calibration of the instrument. Perhaps it wasn't sufficiently equilibrated, or injection errors may have occurred. Otherwise the results are similar to those found for the 0.16 ng/ul concentration. No repeat injections had to be performed and no outliers were detected.

TABLE 4

Summary of Results from MINICAMS Acceptance Testing
for
Instrument 1394

Date Tested	Agent	Sample Size	Calib Conc (ppm)	FAL (TWA)	TAL (TWA)	LOQ (TWA)	Alarm setting 95% confidence level (TWA)
23,24 Jan 91	GB	40	0.16	0.825	0.713	0.112	0.847
30 Jan 91	GB	20	0.055	0.958	0.724	0.176	0.979
23 Jan 91	HD	41	4.8	0.863	0.699	0.150	0.836
31 Jan 91	HD	40	1.62	0.748	0.593	0.352	0.778
24 Jan 91	VX	35	0.016	0.771	0.493	0.478	0.802

FAL = Found Action Level - "is the highest found concentration at which there is a 97.5% level of confidence that the true concentration is less than the hazard-level."*

TAL = Target Action Level - "is the highest true concentration that can be distinguished with a 97.5% level of confidence from hazard-level concentrations." *

LOQ = Limit of Quantitation - "is the lowest true concentration that can be detected at least 97.5% of the time."

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* Hazard-level was defined in the CERTIFY program as 1 TWA equivalent.

TABLE 5

Summary of Results from MINICAMS Acceptance Testing
for
Instrument 1395

Date Tested	Agent	Sample Size	Calib Conc (ppm)	FAL (TWA)	TAL (TWA)	LOQ (TWA)	Alarm setting 95% confidence level (TWA)
23,24 Jan 91	GB	40	0.16	0.794	0.660	0.109	0.821
30 Jan 91	GB	20	0.055	0.995	0.750	0.138	1.014
23 Jan 91	HD	40	4.8	0.824	0.599	0.214	0.853
31 Jan 91	HD	40	1.62	0.780	0.588	0.132	0.814
24 Jan 91	VX	40	0.016	0.875	0.709	0.118	0.893

The same method was used to assess the performance of MINICAMS serial number 1395 for chemical agent GB. Again, two agent standards having concentrations of 0.16 ng/ul and 0.055 ng/ul were used during the testing, with the results being similar to those of instrument number 1394, as can be seen in Table 5. Table 8 and Diagram 4 show the alarm settings and Target vs. Found data, respectively, for the data collected using the 0.055 ng/ul standard. Four repeat injections had to be made during the 0.16 ng/ul trial due to operator error, and five repeat injections were made during the 0.055 ng/ul trial probably due to the bias towards higher concentration readings that was also present in instrument number 1394.

The results for the HD acceptance testing on both instruments mimicked those for the GB acceptance testing as can be seen in Tables 4, 5, 9 and 10, and Diagrams 5 and 6. There seems to be some indication that the instrument alarm set point for chemical agent HD may need to be lowered to ensure the results are within the 95% confidence level. This may further be

determined with additional precision and accuracy studies. Both instruments have successfully demonstrated the ability to detect chemical agents GB and HD when operated in a laboratory environment.

MINICAMS instrument serial number 1394 was challenged with varying quantities of chemical agent VX at a concentration of 0.01[illegible] ng/ul. A total of 47 data points were collected. Of these, 35 were used in the statistical treatment found in Table 11 and Diagram 7, and summarized in Table 4. Of the remaining data points, two were blanks that read 0 TWA, three were spikes at 0.2 TWA whose found concentration was 0 TWA, two were discarded because the operator failed to perform the injection, and the remaining five were not included due to a shift in the retention time of the VX-GB [illegible] necessitating that the agent gate be adjusted. Table 11 shows the alarm settings calculated for this data using the CERTIFY program and Diagram 7 shows the Target vs. Found data for instrument 1394. The small concentration interval between the LOQ and TAL indicates a greater risk of false positives and negatives with this method when analyzing for VX as opposed to GB and HD. Not being able to detect the 0.2 TWA challenge spike is consistent with the high value for the LOQ. The found alarm setting for the 95% confidence level agrees with the manufacturer's recommendation, but with such a limited number of data points used in this statistical treatment of the data, further precision and accuracy studies are recommended to ensure the validity of this number. The uncertainty in the found concentration in the air sampled with this method was determined to be ±25.1% (See Diagram 7), which is on the threshold of being unacceptable from what is required by AMC 385-.31.

VX challenges using 0.023 ng/ul standard along with a 15 minute cycling time on MINICAMS unit 1394 were unsuccessful as indicated by challenge responses frequently being out of acceptance range (Table 1). Data not shown.

The same method was used to assess the performance of MINICAMS instrument serial number 1395 for chemical agent VX. A total of 40 data points were collected and statistically analyzed. The results can be found in Table 12 and Diagram 8 with a brief summary appearing in Table 5. Again in this trial, the problem with variability of the VX-GB analog retention time was encountered, requiring that the operator readjust the agent gate. Also, the 0.2 TWA challenge level was not detected. From Table 5, the large concentration interval between the TAL and LOQ indicates a low risk of false negatives and positives with this method. However, the statistically determined LOQ is in question since the 0.2 TWA challenge level was not capable of being detected. The uncertainty in the analytical results is within the limits set forth by AMC 385-131 (See Diagram 8). As found with instrument 1394, challenges with 0.023 ng/ul VX using a 15 minute cycling time were also unsuccessful on this instrument. Instrument response was frequently out of acceptance range (Table 1). Data not shown.

B. Field Test Evaluation for Agent GB.

Table 6 summarizes the results of testing both instruments in the mobile lab at the test igloo. The resultant alarm settings are shown in Tables 13 and 14 while Diagrams 9 and 10 show the Target vs. Found data. Based on the small concentration interval between the LOQ and TAL for both

instruments, there is a great risk of obtaining false positives and negatives with this analytical technique. Additionally, because the TAL is so close to the LOQ, it would be assumed that keeping an instrument operating at such a high level of performance would be costly regarding maintenance time, spare parts, and frequency of calibration and quality control checks. The found alarm setting for the 95% confidence level was below the alarm setting proposed by the instrument manufacturer. For MINICAMS instrument serial number 1394 with an alarm set at 0.80 TWA, a challenge sample having a GB concentration of 1.00 TWA would cause an instrument alarm only 86.95% of the time (Table 13). For instrument number 1395, the same challenge would result in an alarm 92.03% of the time (Table 14). The uncertainty (with 95% confidence) in the found concentration of GB in an air sample using instrument 1394 was determined to be ±29.97%, which is outside the limit set forth in AMC 385-131 (Diagram 9). For instrument 1395, the uncertainty was determined to be ±26.37% (Diagram 10), also outside the criteria set forth in AMC 385-131.

TABLE 6

Summary of Results from MINICAMS Field Testing
Evaluation for Agent GB

Instrument	Calib Conc (ppm)	Sample Size	FAL (TWA)	TAL (TWA)	LOQ (TWA)	Alarm setting 95% confidence level (TWA)
1394	0.06	290	0.644	0.400	0.279	0.702
1395	0.06	256	0.721	0.472	0.349	0.766

For both instruments some of the initial data sets were rejected because of unsatisfactory instrument calibrations and uncertainty about when the instruments were to be recalibrated. Additional data sets were collected to replace rejected sets. During the test series, erratic data points were excluded from statistical processing when believed that they were tied to an aged preconcentrator tube. Data sets were also excluded if it was apparent that the instrument drifted out of calibration. When an injection was measured outside target limits, it was frequently rerun as a check of operator error. If both measured results were in reasonable agreement, they were both included in the statistical report; otherwise the original injection was recorded but not used in the report.

IV. Discussion.

A. Operator Comments Instrument 1394.

The dominant problem with this unit was with the Relay Preconcentrator (RPC), the frequency of which slowed down testing at the beginning of the Functional Field Test Program. The RPC problem disappeared midway during the test program for no apparent reason. At the beginning of the program we also tried to run 2 single-agent units for additional information. As the single-agent units failed, we were left with only the multiagent units. Possibly, 3 or 4 units created enough current drain on the uninterruptable power supply (UPS) that the UPS output caused the rash of RPC error messages. More probable, the instrument itself was the problem. Since the PCT heater appeared to opera'e well otherwise, the Manufacturer can

probably correct this relay error message easily in some predetermined timing circuit.

No recorder signal could be sent from the MINICAMS unit to its recorder. This should be correctable by the manufacturer or a repair technician.

Flame ignition problems may be caused by excess condensation in the heater block or a poorly flushed hydrogen line and is not deemed serious enough to reflect upon instrument performance.

The instrument was easy to operate and appeared to respond well to agent GB. The MINICAMS unit did not appear to be a problem as a result of van mobility or, with less certainty, a mobile power source.

There is some problem with instrument calibration. The multi-agent software requiring CAL to blink before GB passes through the agent gate is not nearly as user friendly as the older software allowing the operator to calibrate anytime during a cycle. Instrument sensitivity to GB can change during the day although generally the instrument appears stable over days. The instrument may change sensitivity between morning calibration and later field use, but it is felt this is a general instrument condition rather than a problem with stabilization due to time of "warm up". This evaluator is not comfortable with single-point calibration and would recommend more flexibility in calibration and recalibration. Furthermore, there may be a balance of benefits between leaving the instruments on 24 hours/day and shutting them off at night. On the one hand, we have thermal and electrical stability; on the other, the column and PCT heaters are cyclical and would

appear to be subject to greater failure the more they are used.

An interferent peak sometimes elutes immediately after GB and will be detected if the agent gate window is not sufficiently narrow. Changes in nitrogen pressure can cause the peak of interest to shift outside the window.

There appears to be carryover of agent from one cycle to the next when the PCT has been used beyond its useful life. This problem can be overcome by adopting a routine changing interval after the PCT's useful life can be established within tighter bounds than one day to one week. The problem might also be corrected with backflow on the PCT at high temperature to desorb interferences and carryover.

B. Operator Comments Instrument 1395.

There were only two recordings of error codes during the course of field tests, but for about 30 days this unit was not operable. Systematic, step-by-step attempts to determine and correct the problems were unproductive, although with the assistance of service representatives of CMS Research Corp. this evaluator was able to narrow the causes down to computer chips that needed to be replaced and a chip on the interface (I/O) board that was not working.

Repairs of this unit by CMS did in fact involve replacement of the EPROM and BIOS computer chips, and the U13 PIA chip on the I/O board. The NVRAM computer chip was then downloaded with the proper software package. Whatsmore, several ot'er repairs were done to upgrade and stabilize internal parts.

Since installing the repaired unit, there were no error messages or instrument malfunctions in over 65 hours of operating time.

The instrument is easy to operate and did not appear to be adversely affected by truck mobility or use of a mobile power source.

Instrument 1395 was not necessarily calibrated daily because the test plan guidelines said only to calibrate when necessary. Accordingly, on some days the instrument was found to be somewhat out of calibration. Only when it was clear that unit 1395 was miscalibrated, or had drifted out of calibration by a significant degree, was it recalibrated.

Because of an interferent peak that elutes immediately after the GB peak, the agent gate window has to be set very "tightly" around the GB peak which leaves little room for shift in retention time whenever there is a change in nitrogen pressure. A longer chromatographic column would perhaps give better resolution between these peaks, yielding more flexibility in this type of analysis.

Confident determination of which injection to accept for calibration is illusive because of the "single-point" calibration technique. For example, it would be beneficial for the user to be able to input the results from three successive injections and enter the average as the calibration value. Also, the multiagent software requiring CAL to blink before the agent peak passes through the gate is not nearly as user friendly as the single-agent software allowing the operator to calibrate anytime during a cycle.

V. Conclusions.

Both instruments have successfully demonstrated the ability to detect GB and HD when operated in the laboratory. The small concentration interval between the LOQ and TAL for VX on instrument 1394 indicates a high risk of false negatives when sampling at 1 TWA. For the field testing with GB, both instruments had a small interval between LOQ and TAL. In addition, the uncertainty in found concentrations was greater than ± 25% for both instruments. Field testing for HD and VX is still needed before these agents are routinely monitored. Detection of HD in the lab setting looks promising and we expect success in the field. The reliability of VX detection on a routine basis is still in question. It is clear that the operators of these instruments will need thorough maintenance training. Due to a lack of ruggedness in design, if this method is to be incorporated into air monitoring of chemical warfare agents, an abundance of spare parts will be required.

VI. Recommendations.

The MINICAMS as currently configured may meet surveillance requirements for HD. We recommend they be field tested for this agent. Since the instrument did not perform well in the laboratory with VX and considering a recent article in Analytical Chemistry, Vol. 63, pps. 457-459, 1991 by Ketlar et.al. which indicates VX cannot be quantitatively transported through any tubing, we recommend not trying to monitor VX with the MINICAMS mobile laboratory.

Upon examination of the GB field test data, it appears that changing the column packing in the preconcentrator tube and/or changing the liquid phase on the capillary column might enhance the performance of the MINICAMS. Also, allowing for greater operator control over key chromatographic parameters, such as column temperature, hold times, and ramp rates would give each MINICAMS unit the flexibility to be optimized for the particular air environment being sampled, allowing interference peaks to be more readily separated from the agent peak. A backflush arrangement to stop carryover on the preconcentrator tubes is also recommended. If these modifications are made we suggest the data in this report indicate a potential for success in monitoring GB in storage igloos using a MINICAMS mobile laboratory.

Table 7

Alarm settings for GB on instrument 1394 Acceptance Testing using the 0.055 ppm Calibration Std.

PROBABILITY OF ALARM

CONCENTRATION (Z)

INSTRUMENT SETTING	0.2000	0.4000	0.6000	0.8000	1.0000	1.2000	1.4000
0.2	0.4781	0.9997	0.9992	0.9991	0.9899	1.0000	1.0000
0.4	0.0001	0.7119	0.9996	0.9987	1.0000	0.9930	0.9907
0.6	0.0000	0.0033	0.8327	0.9994	0.9997	1.0000	1.0000
0.8	0.0000	0.0000	0.0283	0.8921	0.9993	1.0000	0.9991
1.0	0.0000	0.0000	0.0000	0.1068	0.9234	0.9991	1.0000
1.2	0.0000	0.0000	0.0000	0.0005	0.2357	0.9414	0.9988

ALARM SETTING FOR 95% CONFIDENCE LEVEL = 0.9783
ALARM SETTING FOR 97.5% CONFIDENCE LEVEL = 0.9457
ALARM SETTING FOR 99% CONFIDENCE LEVEL = 0.9059

PROBABILITY OF FALSE ALARM
AT
CONCENTRATION (Z)

INSTRUMENT SETTING	0.1000	0.3000	0.5000	0.7000	0.9000	1.1000
0.05	0.2386	0.0000	0.0000	0.0113	0.0000	0.0000
0.10	0.6773	0.0003	0.0000	0.0074	0.0012	0.0000
0.15	0.9436	0.0020	0.0015	0.0000	0.0105	0.0000
0.20	0.9951	0.0173	0.0000	0.0000	0.0100	0.0012
0.25	0.9997	0.1082	0.0000	0.0000	0.0029	0.0098
0.30	1.0000	0.3886	0.0004	0.0015	0.0000	0.0110
0.35	1.0000	0.7548	0.0019	0.0005	0.0000	0.0060
0.40	1.0000	0.9470	0.0120	0.0000	0.0000	0.0000
0.45	1.0000	0.9928	0.0604	0.0000	0.0014	0.0000
0.50	1.0000	0.9992	0.2169	0.0005	0.0009	0.0000
0.55	1.0000	0.9999	0.5091	0.0021	0.0000	0.0000
0.60	1.0000	1.0000	0.7960	0.0099	0.0000	0.0014
0.65	1.0000	1.0000	0.9445	0.0404	0.0002	0.0011
0.70	1.0000	1.0000	0.9891	0.1326	0.0006	0.0000
0.75	1.0000	1.0000	0.9983	0.3277	0.0024	0.0000
0.80	1.0000	1.0000	0.9997	0.5936	0.0090	0.0000
0.85	1.0000	1.0000	1.0000	0.8191	0.0305	0.0003
0.90	1.0000	1.0000	1.0000	0.9402	0.0900	0.0008
0.95	1.0000	1.0000	1.0000	0.9845	0.2181	0.0027

PROBABILITY OF FALSE ALARM
AT ALARM SETTING OF 0.9457 AND TC OF 0.5 Z = 0.000

TARGET CONCENTRATION AT WHICH PROBABILITY OF FALSE ALARM WITH ALARM SETTING OF 0.9457 IS LESS THAN 5 PERCENT (%) = 0.730

Table 8

Alarm settings for GB on instrument 1395 Acceptance Testing using the 0.055 ppm Calibration Std.

PROBABILITY OF ALARM

CONCENTRATION (Z)

INSTRUMENT SETTING	0.2000	0.4000	0.6000	0.8000	1.0000	1.2000	1.4000
0.2	0.9061	0.9993	0.9990	0.9967	1.0000	1.0000	1.0000
0.4	0.0002	0.9417	1.0000	1.0000	0.9920	0.9954	1.0000
0.6	0.0000	0.0053	0.9548	1.0000	0.9991	1.0000	0.9899
0.8	0.0000	0.0000	0.0420	0.9608	0.9998	0.9988	1.0000
1.0	0.0000	0.0000	0.0000	0.1359	0.9641	0.9997	0.9998
1.2	0.0000	0.0000	0.0000	0.0003	0.2650	0.9660	0.9995

ALARM SETTING FOR 95% CONFIDENCE LEVEL = 1.0138
ALARM SETTING FOR 97.5% CONFIDENCE LEVEL = 0.9853
ALARM SETTING FOR 99% CONFIDENCE LEVEL = 0.9506

PROBABILITY OF FALSE ALARM
AT
CONCENTRATION (Z)

INSTRUMENT SETTING	0.1000	0.3000	0.5000	0.7000	0.9000	1.1000
0.05	0.0065	0.0000	0.0054	0.0000	0.0000	0.0000
0.10	0.1357	0.0015	0.0099	0.0000	0.0000	0.0000
0.15	0.6754	0.0000	0.0000	0.0034	0.0000	0.0000
0.20	0.9734	0.0004	0.0000	0.0114	0.0000	0.0000
0.25	0.9992	0.0058	0.0014	0.0036	0.0034	0.0000
0.30	1.0000	0.0713	0.0000	0.0000	0.0114	0.0000
0.35	1.0000	0.3939	0.0000	0.0000	0.0070	0.0041
0.40	1.0000	0.8309	0.0007	0.0014	0.0000	0.0112
0.45	1.0000	0.9815	0.0066	0.0003	0.0000	0.0088
0.50	1.0000	0.9988	0.0503	0.0000	0.0000	0.0013
0.55	1.0000	0.9999	0.2390	0.0003	0.0015	0.0000
0.60	1.0000	1.0000	0.6067	0.0012	0.0005	0.0000
0.65	1.0000	1.0000	0.8911	0.0079	0.0000	0.0000
0.70	1.0000	1.0000	0.9828	0.0417	0.0000	0.0015
0.75	1.0000	1.0000	0.9981	0.1632	0.0005	0.0006
0.80	1.0000	1.0000	0.9998	0.4257	0.0020	0.0000
0.85	1.0000	1.0000	1.0000	0.7311	0.0092	0.0000
0.90	1.0000	1.0000	1.0000	0.9178	0.0373	0.0003
0.95	1.0000	1.0000	1.0000	0.9824	0.1232	0.0008

PROBABILITY OF FALSE ALARM
AT ALARM SETTING OF 0.9853 AND TC OF 0.5 Z = 0.000

TARGET CONCENTRATION AT WHICH PROBABILITY OF FALSE ALARM WITH ALARM SETTING OF 0.9853 IS LESS THAN 5 PERCENT (%) = 0.755

Table 9
Alarm settings for HD on instrument 1394 Acceptance Testing using the 1.62 ppm Calibration Std.

PROBABILITY OF ALARM

CONCENTRATION (Z)

INSTRUMENT SETTING	0.2000	0.4000	0.6000	0.8000	1.0000	1.2000	1.4000
0.2	0.2872	0.9609	0.9997	1.0000	0.9991	1.0000	1.0000
0.4	0.0020	0.3203	0.9513	0.9994	1.0000	0.9994	1.0000
0.6	0.0000	0.0045	0.3514	0.9403	0.9988	1.0000	1.0000
0.8	0.0000	0.0000	0.0090	0.3794	0.9283	0.9977	0.9999
1.0	0.0000	0.0000	0.0000	0.0165	0.4040	0.9160	0.9960
1.2	0.0000	0.0000	0.0000	0.0001	0.0272	0.4253	0.9036

ALARM SETTING FOR 95% CONFIDENCE LEVEL = 0.7780
ALARM SETTING FOR 97.5% CONFIDENCE LEVEL = 0.7391
ALARM SETTING FOR 99% CONFIDENCE LEVEL = 0.6928

PROBABILITY OF FALSE ALARM
AT
CONCENTRATION (Z)

INSTRUMENT SETTING	0.1000	0.3000	0.5000	0.7000	0.9000	1.1000
0.05	0.4891	0.0092	0.0000	0.0005	0.0000	0.0000
0.10	0.7295	0.0350	0.0002	0.0000	0.0005	0.0000
0.15	0.8926	0.1062	0.0008	0.0000	0.0009	0.0000
0.20	0.9680	0.2526	0.0035	0.0000	0.0004	0.0000
0.25	0.9926	0.4693	0.0134	0.0001	0.0000	0.0007
0.30	0.9986	0.6961	0.0436	0.0004	0.0000	0.0008
0.35	0.9998	0.8637	0.1171	0.0016	0.0000	0.0004
0.40	1.0000	0.9522	0.2554	0.0059	0.0001	0.0000
0.45	1.0000	0.9867	0.4531	0.0192	0.0003	0.0000
0.50	1.0000	0.9970	0.6638	0.0541	0.0009	0.0000
0.55	1.0000	0.9994	0.8313	0.1292	0.0030	0.0001
0.60	1.0000	0.9999	0.9313	0.2600	0.0094	0.0002
0.65	1.0000	1.0000	0.9770	0.4400	0.0265	0.0005
0.70	1.0000	1.0000	0.9936	0.6342	0.0656	0.0017
0.75	1.0000	1.0000	0.9985	0.7978	0.1419	0.0051
0.80	1.0000	1.0000	0.9997	0.9061	0.2653	0 0140
0.85	1.0000	1.0000	0.9999	0.9632	0.4297	0.0349
0.90	1.0000	1.0000	1.0000	0.9877	0.6079	0.0778
0.95	1.0000	1.0000	1.0000	0.9964	0.7651	0.1543

PROBABILITY OF FALSE ALARM
AT ALARM SETTING OF 0.7391 AND TC OF 0.5 Z = 0.002

TARGET CONCENTRATION AT WHICH PROBABILITY OF FALSE ALARM WITH ALARM SETTING OF 0.7391 IS LESS THAN 5 PERCENT (%) = 0.613

Table 10
Alarm settings for HD on instrument 1395 Acceptance Testing using the 1.62 ppm Calibration Std.

PROBABILITY OF ALARM

CONCENTRATION (Z)

INSTRUMENT SETTING	0.2000	0.4000	0.6000	0.8000	1.0000	1.2000	1.4000
0.2	0.9559	1.0000	1.0000	0.9998	0.9996	0.9995	0.9994
0.4	0.0000	0.7993	0.9982	0.9999	1.0000	1.0000	1.0000
0.6	0.0000	0.0011	0.7127	0.9853	0.9987	0.9997	0.9999
0.8	0.0000	0.0000	0.0280	0.6680	0.9589	0.9941	0.9988
1.0	0.0000	0.0000	0.0000	0.0873	0.6416	0.9266	0.9850
1.2	0.0000	0.0000	0.0000	0.0013	0.1494	0.6243	0.8941

ALARM SETTING FOR 95% CONFIDENCE LEVEL = 0.8140
ALARM SETTING FOR 97.5% CONFIDENCE LEVEL = 0.7664
ALARM SETTING FOR 99% CONFIDENCE LEVEL = 0.7097

PROBABILITY OF FALSE ALARM AT CONCENTRATION (Z)

INSTRUMENT SETTING	0.1000	0.3000	0.5000	0.7000	0.9000	1.1000
0.05	0.0000	0.0000	0.0000	0.0000	0.0000	0.0004
0.10	0.0156	0.0005	0.0005	0.0007	0.0008	0.0008
0.15	0.8026	0.0000	0.0006	0.0007	0.0008	0.0008
0.20	0.9998	0.0001	0.0000	0.0001	0.0003	0.0004
0.25	1.0000	0.0062	0.0000	0.0000	0.0000	0.0001
0.30	1.0000	0.1273	0.0003	0.0000	0.0000	0.0000
0.35	1.0000	0.6195	0.0021	0.0001	0.0000	0.0000
0.40	1.0000	0.9577	0.0152	0.0004	0.0001	0.0000
0.45	1.0000	0.9988	0.0760	0.0016	0.0002	0.0000
0.50	1.0000	1.0000	0.2518	0.0063	0.0005	0.0001
0.55	1.0000	1.0000	0.5438	0.0220	0.0014	0.0002
0.60	1.0000	1.0000	0.8122	0.0644	0.0040	0.0005
0.65	1.0000	1.0000	0.9498	0.1566	0.0108	0.0013
0.70	1.0000	1.0000	0.9910	0.3129	0.0267	0.0031
0.75	1.0000	1.0000	0.9988	0.5152	0.0595	0.0069
0.80	1.0000	1.0000	0.9999	0.7134	0.1197	0.0148
0.85	1.0000	1.0000	1.0000	0.8606	0.2155	0.0300
0.90	1.0000	1.0000	1.0000	0.9443	0.3467	0.0569
0.95	1.0000	1.0000	1.0000	0.9815	0.5006	0.1010

PROBABILITY OF FALSE ALARM
AT ALARM SETTING OF 0.7664 AND TC OF 0.5 Z = 0.001

TARGET CONCENTRATION AT WHICH PROBABILITY OF FALSE ALARM WITH ALARM SETTING OF 0.7664 IS LESS THAN 5 PERCENT (%) = 0.591

Table 11
Alarm settings for VX on instrument 1394

PROBABILITY OF ALARM

CONCENTRATION (Z)

INSTRUMENT SETTING	0.2000	0.4000	0.6000	0.8000	1.0000	1.2000	1.4000
0.2	0.8347	0.9945	0.9999	1.0000	0.9990	1.0000	1.0000
0.4	0.1499	0.7640	0.9875	0.9997	1.0000	0.9995	0.9994
0.6	0.0019	0.1132	0.6834	0.9741	0.9990	1.0000	1.0000
0.8	0.0000	0.0015	0.0881	0.6004	0.9515	0.9973	0.9998
1.0	0.0000	0.0000	0.0013	0.0712	0.5218	0.9184	0.9936
1.2	0.0000	0.0000	0.0000	0.0013	0.0598	0.4515	0.8753

ALARM SETTING FOR 95% CONFIDENCE LEVEL = 0.8020
ALARM SETTING FOR 97.5% CONFIDENCE LEVEL = 0.7607
ALARM SETTING FOR 99% CONFIDENCE LEVEL = 0.7113

PROBABILITY OF FALSE ALARM
AT
CONCENTRATION (Z)

INSTRUMENT SETTING	0.1000	0.3000	0.5000	0.7000	0.9000	1.1000
0.05	0.0554	0.0010	0.0000	0.0007	0.0000	0.0000
0.10	0.1354	0.0036	0.0001	0.0001	0.0006	0.0000
0.15	0.2759	0.0121	0.0002	0.0000	0.0010	0.0000
0.20	0.4671	0.0359	0.0007	0.0000	0.0005	0.0001
0.25	0.6667	0.0919	0.0025	0.0000	0.0000	0.0009
0.30	0.8262	0.1990	0.0084	0.0002	0.0000	0.0008
0.35	0.9247	0.3622	0.0247	0.0006	0.0000	0.0003
0.40	0.9726	0.5572	0.0641	0.0020	0.0001	0.0000
0.45	0.9915	0.7386	0.1438	0.0063	0.0002	0.0000
0.50	0.9977	0.8702	0.2755	0.0182	0.0006	0.0000
0.55	0.9994	0.9457	0.4510	0.0468	0.0017	0.0001
0.60	0.9999	0.9806	0.6372	0.1061	0.0052	0.0002
0.65	1.0000	0.9940	0.7944	0.2093	0.0143	0.0006
0.70	1.0000	0.9983	0.9006	0.3583	0.0359	0.0016
0.75	1.0000	0.9996	0.9587	0.5342	0.0807	0.0046
0.80	1.0000	0.9999	0.9851	0.7033	0.1610	0.0120
0.85	1.0000	1.0000	0.9952	0.835[illegible]	0.2833	0.0289
0.90	1.0000	1.0000	0.9986	0.92[illegible]	0.4398	0.0637
0.95	1.0000	1.0000	0.9996	0.9669	0.6069	0.1265

PROBABILITY OF FALSE ALARM
AT ALARM SETTING OF 0.7607 AND TC OF 0.5 Z = 0.034

TARGET CONCENTRATION AT WHICH PROBABILITY OF FALSE ALARM WITH ALARM SETTING OF 0.7607 IS LESS THAN 5 PERCENT (%) = 0.522

Table 12
Alarm settings for VX on instrument 1395

PROBABILITY OF ALARM

CONCENTRATION (Z)

INSTRUMENT SETTING	0.2000	0.4000	0.6000	0.8000	1.0000	1.2000	1.4000
0.2	0.9970	1.0000	1.0000	0.9941	0.9908	0.9898	0.9898
0.4	0.9488	0.9997	1.0000	0.9943	0.9916	0.9974	1.0000
0.6	0.0000	0.1974	0.9975	1.0000	0.9992	1.0000	1.0000
0.8	0.0000	0.0000	0.0139	0.8562	0.9990	1.0000	0.9996
1.0	0.0000	0.0000	0.0000	0.0016	0.4272	0.9678	0.9993
1.2	0.0000	0.0000	0.0000	0.0000	0.0003	0.1438	0.8008

ALARM SETTING FOR 95% CONFIDENCE LEVEL = 0.8930
ALARM SETTING FOR 97.5% CONFIDENCE LEVEL = 0.8737
ALARM SETTING FOR 99% CONFIDENCE LEVEL = 0.8507

PROBABILITY OF FALSE ALARM
AT
CONCENTRATION (Z)

INSTRUMENT SETTING	0.1000	0.3000	0.5000	0.7000	0.9000	1.1000
0.05	0.0110	0.0000	0.0000	0.0000	0.0069	0.0268
0.10	0.0150	0.0351	0.0283	0.0369	0.0440	0.0472
0.15	0.0000	0.0184	0.0402	0.0421	0.0406	0.0380
0.20	0.0000	0.0000	0.0000	0.0025	0.0080	0.0099
0.25	0.0005	0.0089	0.0000	0.0000	0.0000	0.0000
0.30	0.0006	0.0000	0.0087	0.0000	0.0000	0.0000
0.35	0.1841	0.0005	0.0000	0.0089	0.0016	0.0000
0.40	0.9512	0.0001	0.0000	0.0003	0.0086	0.0060
0.45	0.9999	0.0170	0.0001	0.0000	0.0007	0.0079
0.50	1.0000	0.4120	0.0001	0.0009	0.0000	0.0007
0.55	1.0000	0.9560	0.0040	0.0000	0.0003	0.0000
0.60	1.0000	0.9997	0.0995	0.0001	0.0005	0.0000
0.65	1.0000	1.0000	0.5717	0.0018	0.0000	0.0009
0.70	1.0000	1.0000	0.9486	0.0310	0.0001	0.0001
0.75	1.0000	1.0000	0.9985	0.2286	0.0012	0.0000
0.80	1.0000	1.0000	1.0000	0.6611	0.0134	0.0001
0.85	1.0000	1.0000	1.0000	0.9399	0.0934	0.0009
0.90	1.0000	1.0000	1.0000	0.9956	0.3500	0.0074
0.95	1.0000	1.0000	1.0000	0.9998	0.7130	0.0443

PROBABILITY OF FALSE ALARM
AT ALARM SETTING OF 0.8737 AND TC OF 0.5 Z = 0.000

TARGET CONCENTRATION AT WHICH PROBABILITY OF FALSE ALARM WITH ALARM SETTING OF 0.8737 IS LESS THAN 5 PERCENT (%) = 0.725

Table 13

Alarm settings for GB on instrument 1394, for Field testing

PROBABILITY OF ALARM

CONCENTRATION (Z)

INSTRUMENT SETTING	0.2000	0.4000	0.6000	0.8000	1.0000	1.2000	1.4000
0.2	0.7658	0.9910	0.9995	0.9999	1.0000	1.0000	1.0000
0.4	0.0259	0.6625	0.9623	0.9960	0.9994	0.9999	1.0000
0.6	0.0000	0.0623	0.5957	0.9183	0.9859	0.9973	0.9994
0.8	0.0000	0.0003	0.0973	0.5506	0.8695	0.9683	0.9922
1.0	0.0000	0.0000	0.0024	0.1270	0.5186	0.8222	0.9447
1.2	0.0000	0.0000	0.0000	0.0082	0.1513	0.4949	0.7793

ALARM SETTING FOR 95% CONFIDENCE LEVEL = 0.7029
ALARM SETTING FOR 97.5% CONFIDENCE LEVEL = 0.6439
ALARM SETTING FOR 99% CONFIDENCE LEVEL = 0.5751

PROBABILITY OF FALSE ALARM
AT
CONCENTRATION (Z)

INSTRUMENT SETTING	0.1000	0.3000	0.5000	0.7000	0.9000	1.1000
0.05	0.0368	0.0004	0.0000	0.0000	0.0000	0.0000
0.10	0.1642	0.0026	0.0001	0.0000	0.0000	0.0000
0.15	0.4358	0.0127	0.0005	0.0000	0.0000	0.0000
0.20	0.7436	0.0468	0.0019	0.0002	0.0000	0.0000
0.25	0.9290	0.1328	0.0069	0.0005	0.0001	0.0000
0.30	0.9886	0.2916	0.0208	0.0016	0.0002	0.0000
0.35	0.9990	0.5067	0.0537	0.0047	0.0006	0.0001
0.40	0.9999	0.7199	0.1187	0.0120	0.0015	0.0002
0.45	1.0000	0.8743	0.2263	0.0279	0.0036	0.0006
0.50	1.0000	0.9563	0.3744	0.0585	0.0081	0.0013
0.55	1.0000	0.9884	0.5442	0.1109	0.0172	0.0029
0.60	1.0000	0.9976	0.7062	0.1909	0.0338	0.0061
0.65	1.0000	0.9996	0.8347	0.2991	0.0620	0.0119
0.70	1.0000	1.0000	0.9196	0.4289	0.1060	0.0220
0.75	1.0000	1.0000	0.9664	0.5671	0.1692	0.0387
0.80	1.0000	1.0000	0.9880	0.6974	0.2526	0.0647
0.85	1.0000	1.0000	0.9963	0.8063	0.3538	0.1026
0.90	1.0000	1.0000	0.9990	0.8871	0.4667	0.1546
0.95	1.0000	1.0000	0.9998	0.9403	0.5824	0.2216

PROBABILITY OF FALSE ALARM
AT ALARM SETTING OF 0.6439 AND TC OF 0.5 Z = 0.179

TARGET CONCENTRATION AT WHICH PROBABILITY OF FALSE ALARM WITH ALARM SETTING OF 0.6439 IS LESS THAN 5 PERCENT (%) = 0.427

Table 14
Alarm settings for GB on instrument 1395 for Field Testing

PROBABILITY OF ALARM

CONCENTRATION (Z)

INSTRUMENT SETTING	0.2000	0.4000	0.6000	0.8000	1.0000	1.2000	1.4000
0.2	0.8274	0.9962	0.9999	1.0000	1.0000	1.0000	1.0000
0.4	0.0662	0.7265	0.9839	0.9995	1.0000	1.0000	1.0000
0.6	0.0000	0.0696	0.6359	0.9583	0.9974	0.9999	1.0000
0.8	0.0000	0.0002	0.0732	0.5608	0.9203	0.9918	0.9993
1.0	0.0000	0.0000	0.0006	0.0767	0.5002	0.8740	0.9810
1.2	0.0000	0.0000	0.0000	0.0014	0.0798	0.4516	0.8239

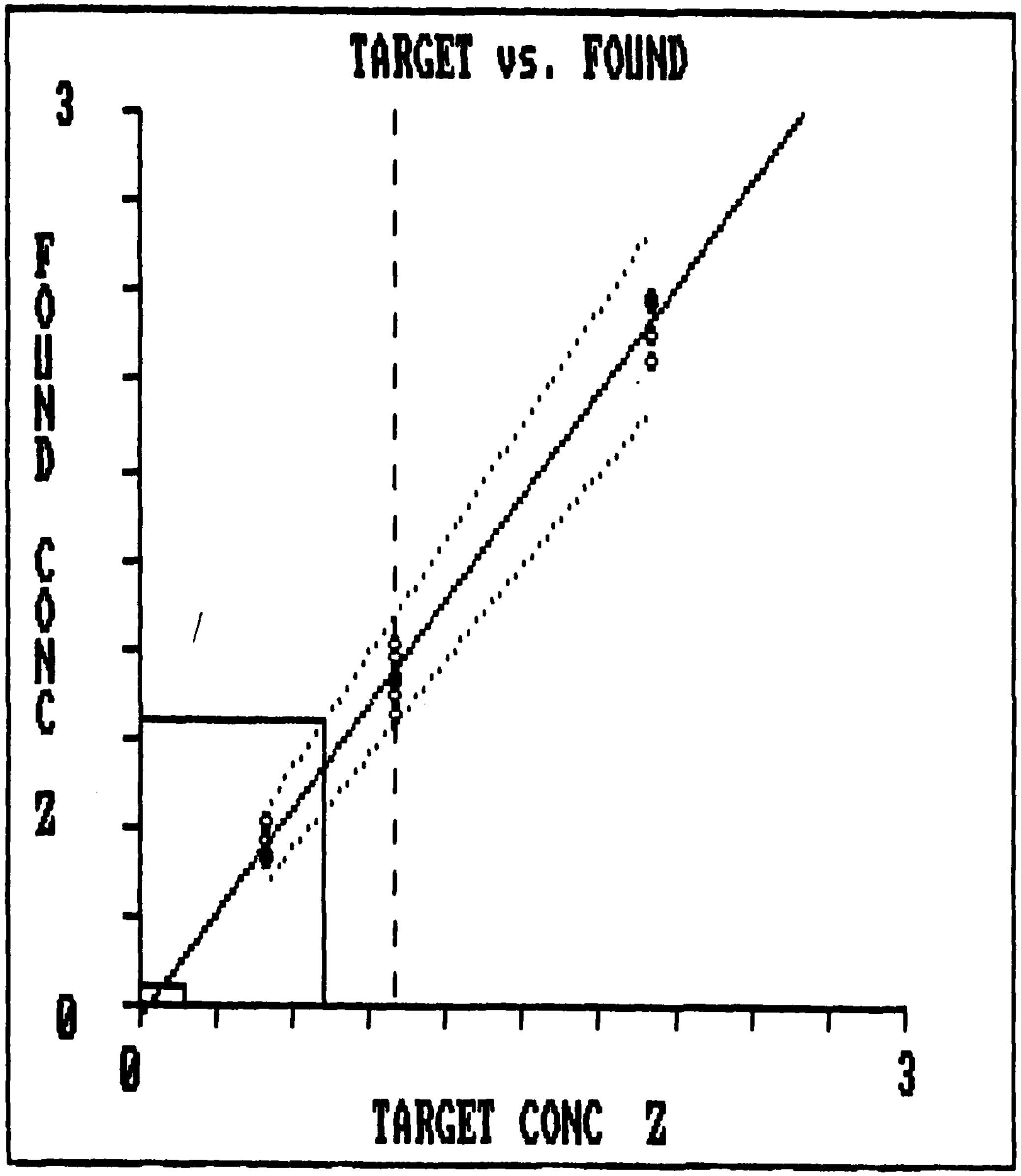
ALARM SETTING FOR 95% CONFIDENCE LEVEL = 0.7660
ALARM SETTING FOR 97.5% CONFIDENCE LEVEL = 0.7208
ALARM SETTING FOR 99% CONFIDENCE LEVEL = 0.6682

PROBABILITY OF FALSE ALARM
AT
CONCENTRATION (Z)

INSTRUMENT SETTING	0.1000	0.3000	0.5000	0.7000	0.9000	1.1000
0.05	0.0340	0.0002	0.0000	0.0000	0.0000	0.0000
0.10	0.1232	0.0014	0.0000	0.0000	0.0000	0.0000
0.15	0.3117	0.0074	0.0001	0.0000	0.0000	0.0000
0.20	0.5709	0.0299	0.0004	0.0000	0.0000	0.0000
0.25	0.8017	0.0931	0.0021	0.0000	0.0000	0.0000
0.30	0.9351	0.2236	0.0085	0.0002	0.0000	0.0000
0.35	0.9853	0.4221	0.0281	0.0008	0.0000	0.0000
0.40	0.9977	0.6433	0.0763	0.0029	0.0001	0.0000
0.45	0.9998	0.8240	0.1714	0.0096	0.0004	0.0000
0.50	1.0000	0.9322	0.3206	0.0272	0.0012	0.0001
0.55	1.0000	0.9799	0.5070	0.0661	0.0039	0.0002
0.60	1.0000	0.9954	0.6918	0.1387	0.0108	0.0006
0.65	1.0000	0.9992	0.8374	0.2529	0.0268	0.0018
0.70	1.0000	0.9999	0.9286	0.4036	0.0593	0.0048
0.75	1.0000	1.0000	0.9741	0.5705	0.1171	0.0118
0.80	1.0000	1.0000	0.9923	0.7254	0.2068	0.0267
0.85	1.0000	1.0000	0.9981	0.8461	0.3281	0.0546
0.90	1.0000	1.0000	0.9996	0.9250	0.4712	0.1021
0.95	1.0000	1.0000	0.9999	0.9684	0.6181	0.1744

PROBABILITY OF FALSE ALARM
AT ALARM SETTING OF 0.7208 AND TC OF 0.5 Z = 0.048

TARGET CONCENTRATION AT WHICH PROBABILITY OF FALSE ALARM WITH ALARM SETTING OF 0.7208 IS LESS THAN 5 PERCENT (%) = 0.502

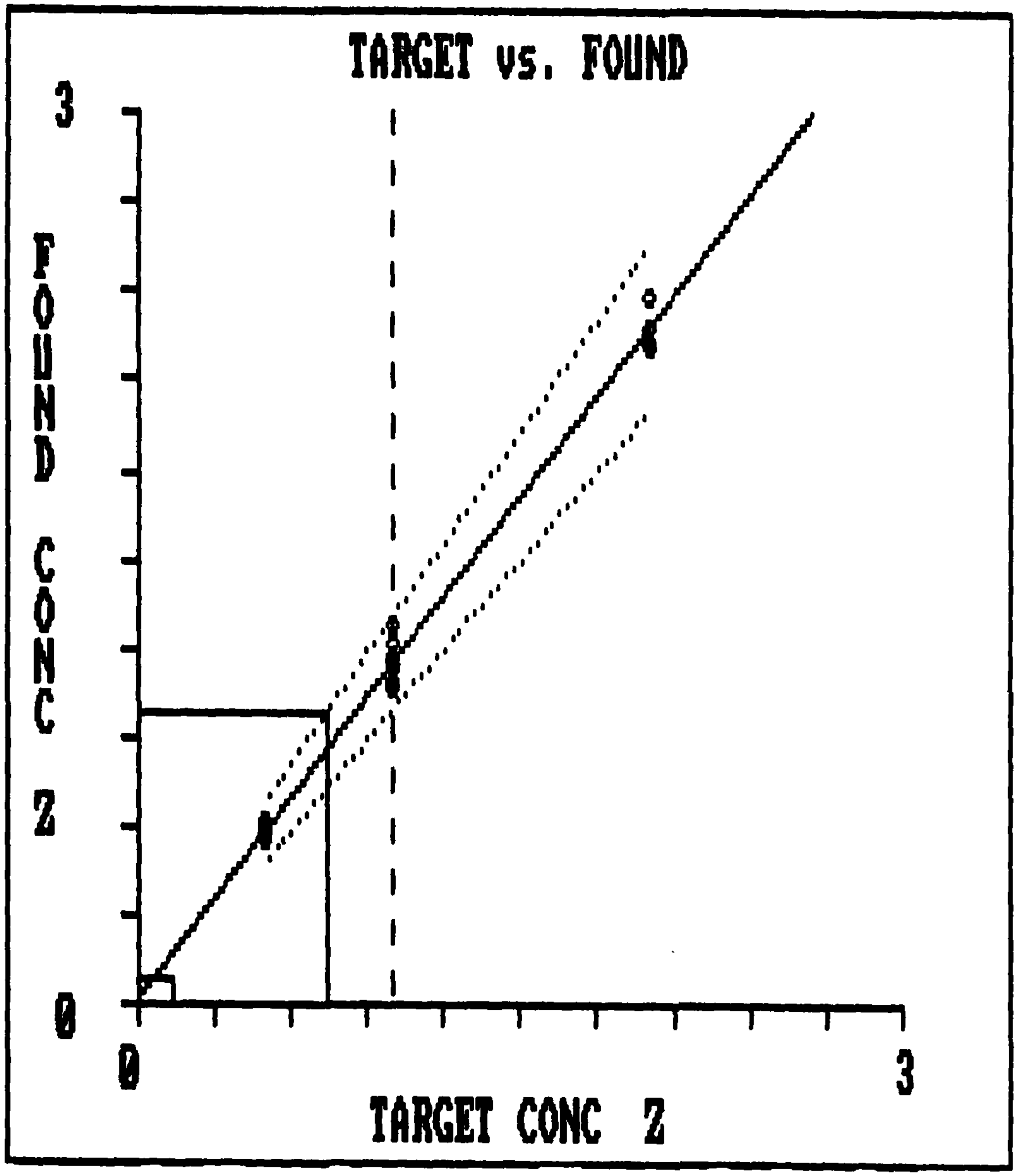


% UIAS (±)	=	13.7125
FAL	=	.9580666
TAL	=	.7249756
LOQ	=	.1760253
DECIS	=	.0642813
% RECOVERY	=	113.3831
DATA POINTS	=	20
SLOPE	=	1.170478
INTERCEPT	=	-.0366474
% UIFM (±)	=	14.90773
Z	=	1

AGENT: GB LAB: SMCPB-QAL DATES: 30 JAN 91

Diagram 3

Target vs. Found Data for GB on Instrument 1394 Acceptance Tesing using the 0.055 ppm Calibration Standard

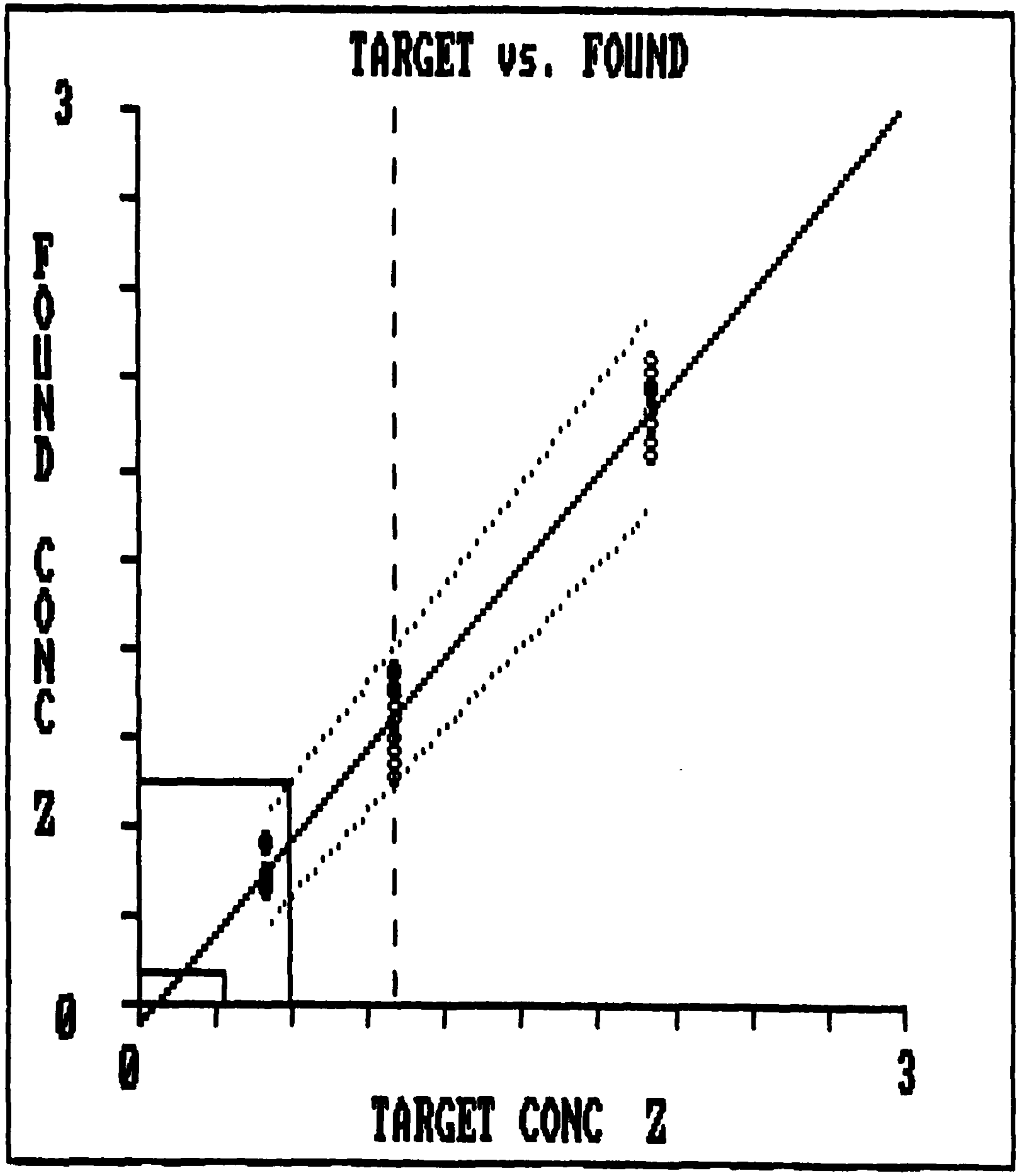


% UIAS (±)	=	12.5012
FAL	=	.9947479
TAL	=	.7495117
LOQ	=	.1381836
DECIS	=	.099249
% RECOVERY	=	114.9667
DATA POINTS	=	20
SLOPE	=	1.126846
INTERCEPT	=	.0228204
% UIFM (±)	=	13.02767
Z	=	1

AGENT: GB LAB: SMCPB-QAL DATES: 30 JAN 91

Diagram 4

Target vs. Found Data for GB on Instrument 1395 Acceptance Testing using the 0.055 ppm Calibration Standard

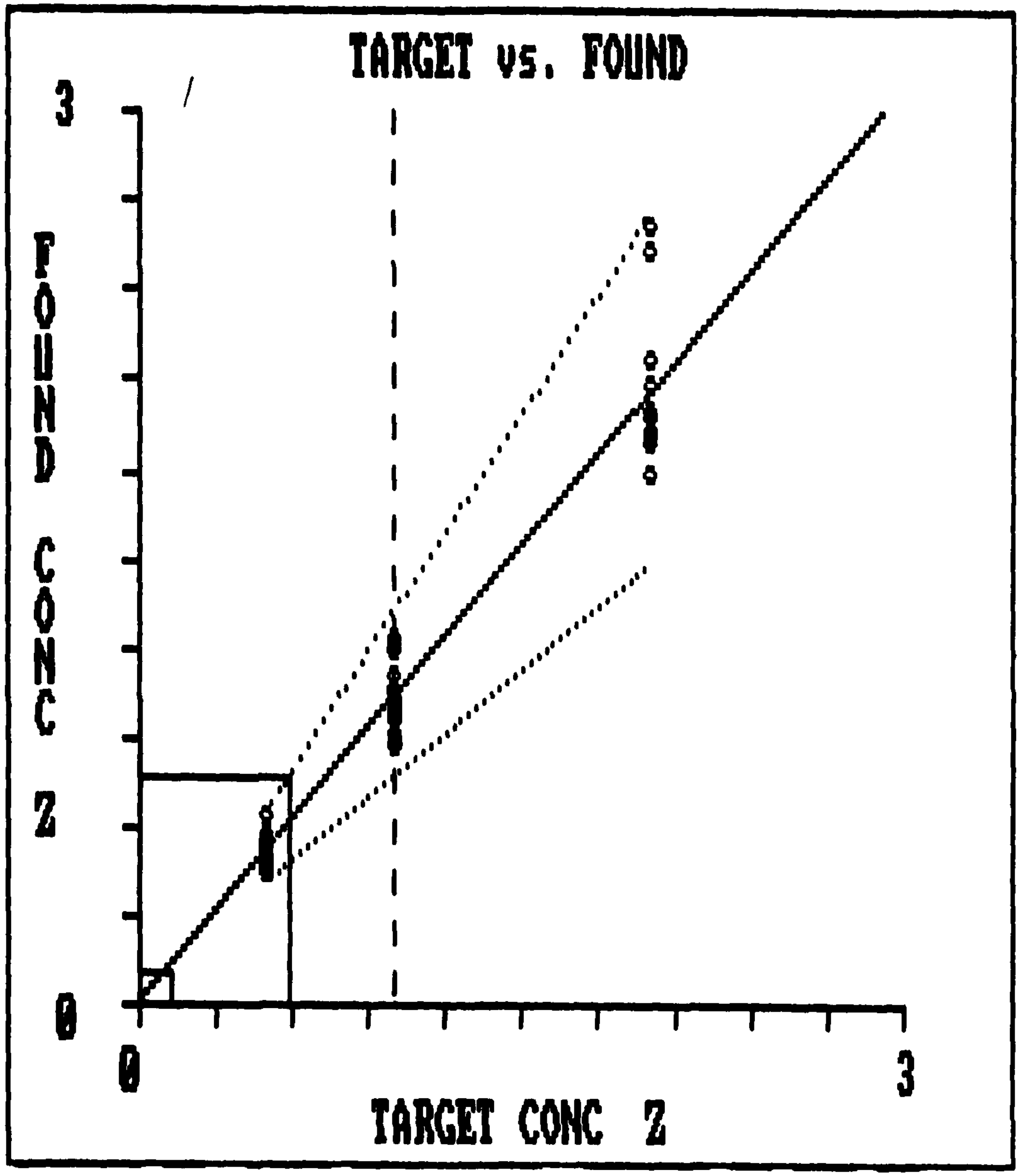


% UIAS (±)	=	20.21446
FAL	=	.7481527
TAL	=	.59375
LOQ	=	.3327636
DECIS	=	.1141332
% RECOVERY	=	97.18325
DATA POINTS	=	40
SLOPE	=	1.021887
INTERCEPT	=	-.0500541
% UIFM (±)	=	22.12701
Z	=	1

AGENT: HD LAB: SMCPB-QAL DATES: 31 JAN 91

Diagram 5

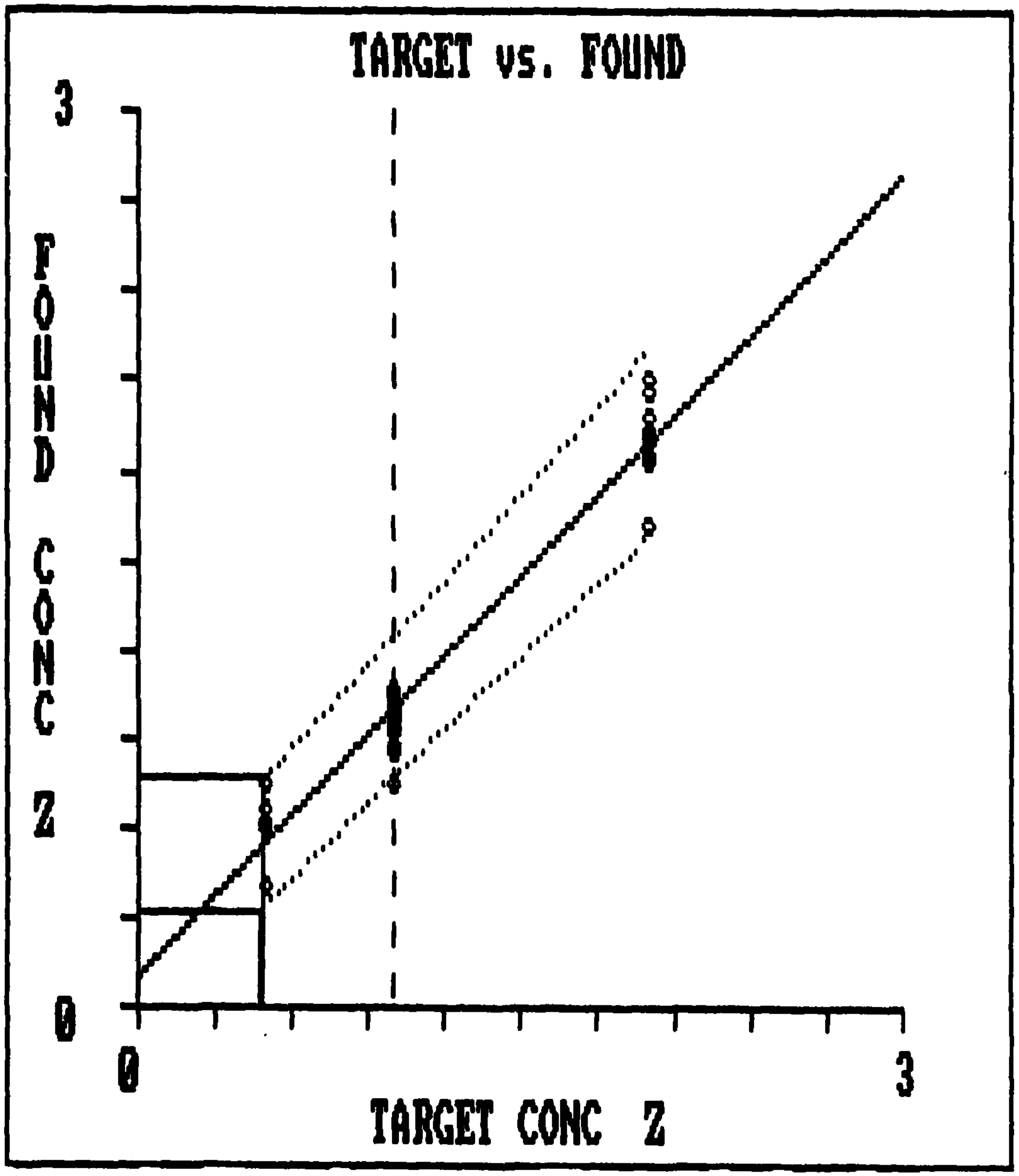
Target vs. Found Data for HD Testing on Instrument 1394 using the 1.52 ppm Calibration Standard



% UIAS (±)	=	20.51853
FAL	=	.7796628
TAL	=	.5883789
LOQ	=	.1323242
DECIS	=	.1111622
% RECOUERY	=	105.1527
DATA POINTS	=	40
SLOPE	=	1.01714
INTERCEPT	=	.0343866
% UIFM (±)	=	20.84743
Z	=	1

AGENT: HD LAB: SMCPB-QAL DATES: 31 JAN 91

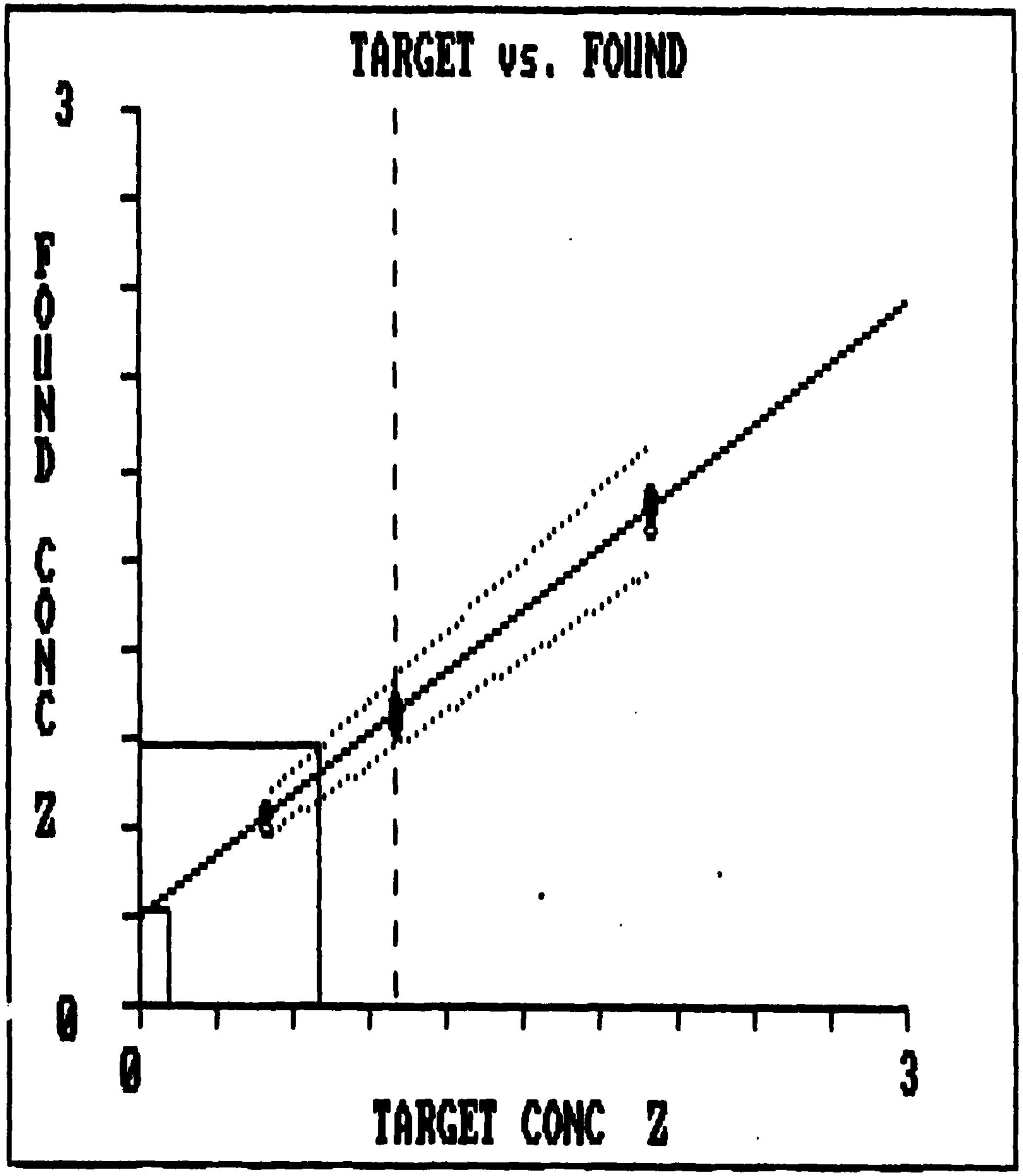
Diagram 6
Target vs. Found Data for HD Testing on Instrument 1395 using sthe 1.62 ppm Calibration Standard



% UIAS (±)	=	25.11594
FAL	=	.7710867
TAL	=	.493164
LOQ	=	.4777832
DECIS	=	.3294602
% RECOVERY	=	100.6656
DATA POINTS	=	35
SLOPE	=	.8873516
INTERCEPT	=	.1193045
% UIFM (±)	=	22.97854
Z	=	1

AGENT: VX LAB: SMCPB-QAL DATES: 24 JAN 91

Diagram 7
Target vs. Found Data for VX Testing on Instrument 1394

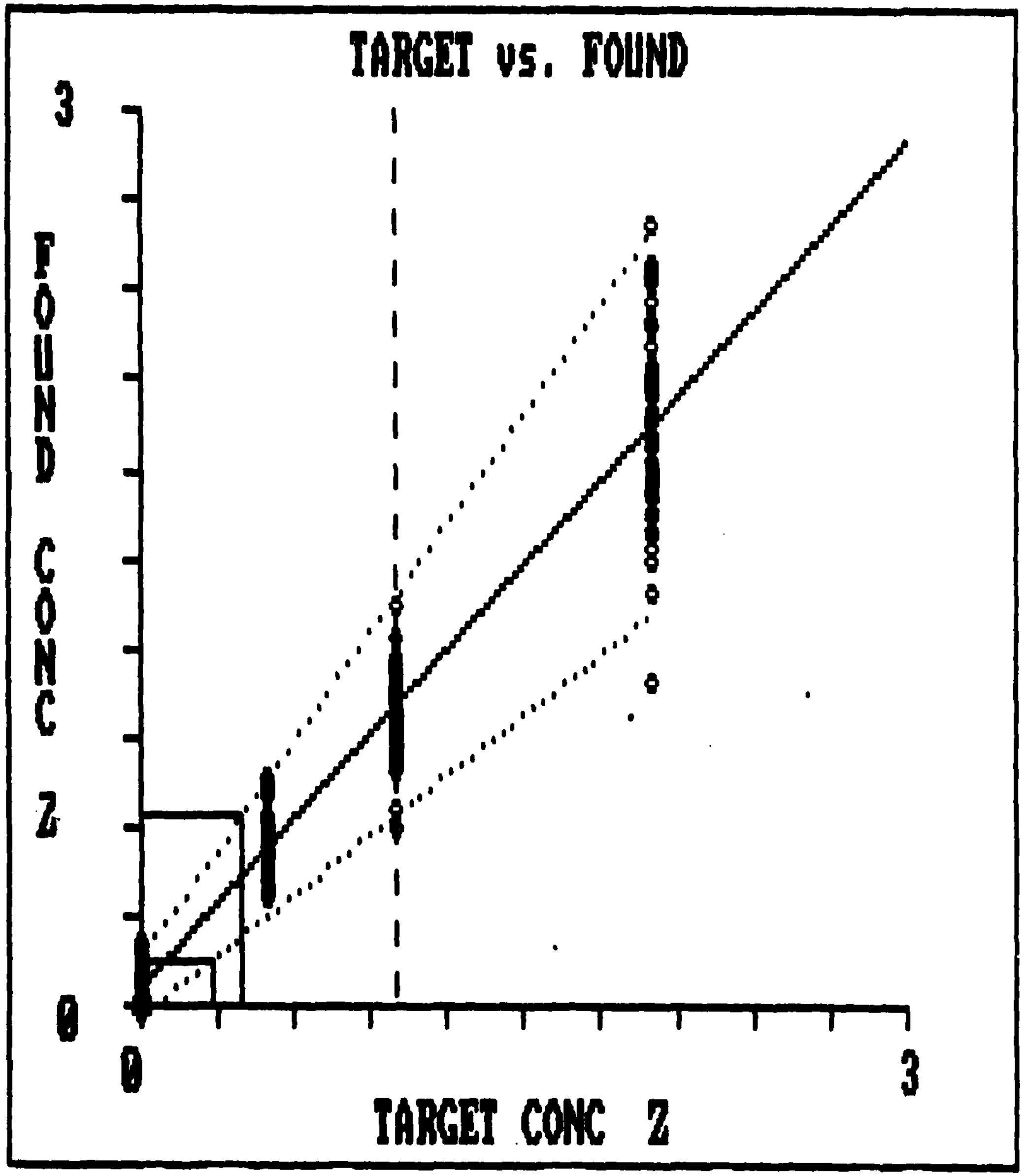


% VIAS (±)	=	14.49649
FAL	=	.875093
TAL	=	.7094726
LOQ	=	.1179199
DECIS	=	.3376665
% RECOVERY	=	98.94273
DATA POINTS	=	40
SLOPE	=	.6910733
INTERCEPT	=	.2983539
% VIFM (±)	=	11.43469
Z	=	1

AGENT: VX LAB: SMCPB-QAL DATES: 24 JAN 91

Diagram 8

Target vs.Found Data for VX Testing on Instrument 1395



% UIAS (±) = 29.96842

FAL = .643934

TAL = .4002685

LOQ = .2785644

DECIS = .1602888

% RECOVERY = 100.8646

DATA POINTS = 290

SLOPE = .943051

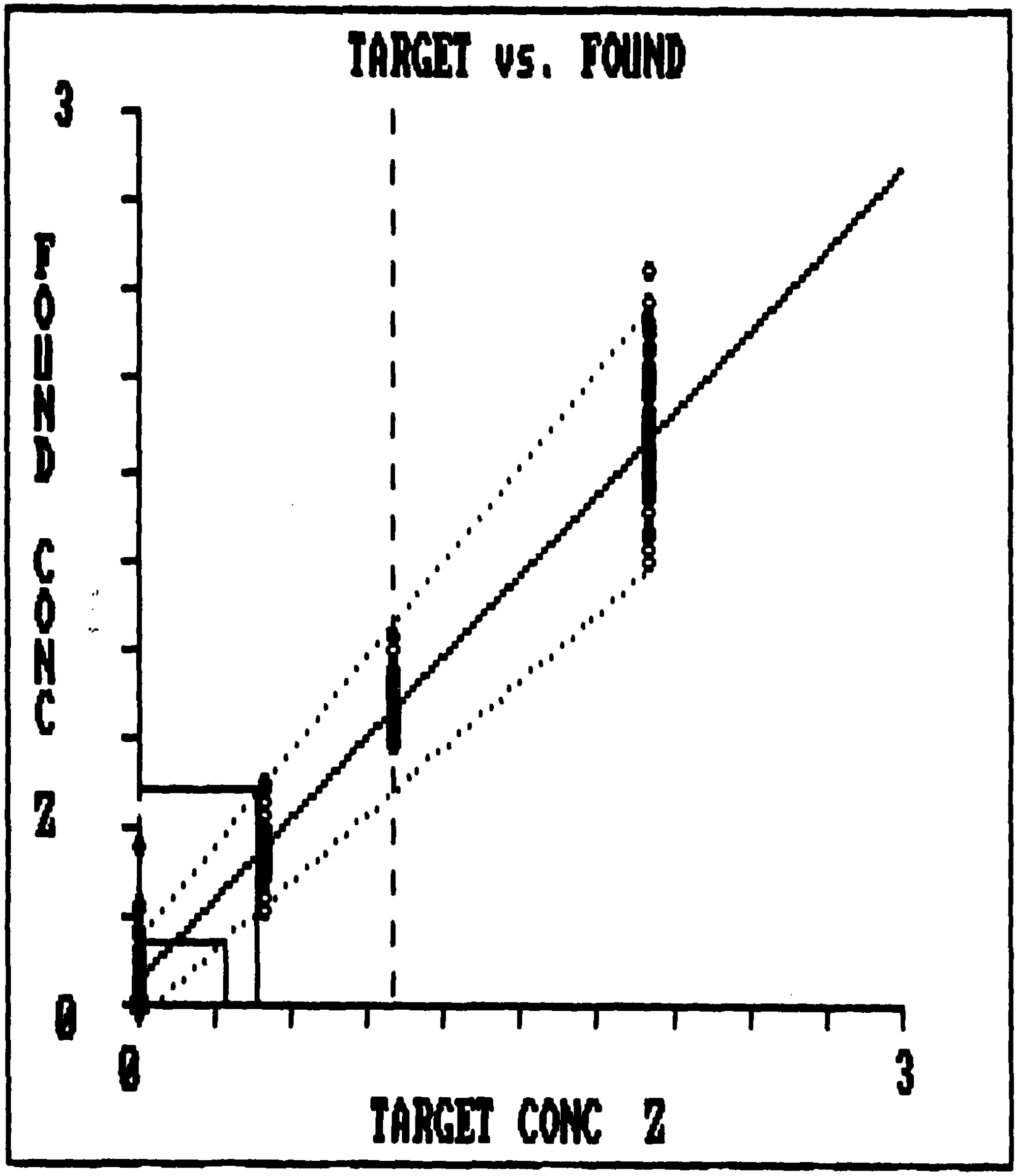
INTERCEPT = .0655947

% UIFM (±) = 28.92329

Z = 1

AGENT: GB LAB: SMCPB-QAL DATES: 3/5/91 - 4/1/91

Diagram 9
Target vs. Found Data for GB Field Testing on Instrument 1394



% UIAS (±)	=	26.3689
FAL	=	.7214458
TAL	=	.4715576
LOQ	=	.3491211
DECIS	=	.230523
% RECOVERY	=	100.0074
DATA POINTS	=	256
SLOPE	=	.9037855
INTERCEPT	=	.0962887
% UIFM (±)	=	24.7744
Z	=	1

AGENT: GB LAB: SMCPB-QAL DATES: 7-13 Mar & 16-24 Apr 91

Diagram 10
Target vs. Found Data for GB Field Testing on Instrument 1395

Appendix 1

DETAILED TEST PLAN

FOR THE

FUNCTIONAL FIELD TEST

OF THE

MINI - CHEMICAL AGENT MONITOR FIRST ENTRY MONITORING SYSTEM

JANUARY 1990

TEST & EVALUATION OFFICE
RESEARCH, DEVELOPMENT & ENGINEERING SUPPORT DIRECTORATE
CHEMICAL RESEARCH DEVELOPMENT AND ENGINEERING CENTER
ABERDEEN PROVING GROUND, MD 21010-5423

CONTENTS

Subject

1.1 BACKGROUND. First entry monitoring of chemical storage depots currently consists of incorporating a combination of bubblers, blue band detector tubes, and M8 automatic chemical detectors. These methods are time consuming, expensive and labor intensive. A potential solution to upgrade current first entry monitoring procedures is to use off the shelf laboratory chemical detection equipment. A system consisting of a Mini - Chemical Agent Monitor (MINICAM) mounted in a "laboratory" type vehicle with a heated tubs that could be "plugged" into a chemical munition storage igloo is being considered as a replacement for current first entry monitoring procedures. The primary concern with this approach is that the MINICAM is designed to be used in a controlled laboratory environment and therfore may not function properly when subjected to vibrations encountered while operating in a moving vehicle. This test program is being conducted to determine the ability of the proposed system to conduct first entry monitoring.

1.2 SYSTEM DESCRIPTION. The system to be tested will consist of a MINICAM shock mounted in a "laboratory" type vehicle with a 75 foot heated tube connected to a vaccum pump to obtain a potential vapor agent sample from the storage igloo.

1.3 TEST OBJECTIVE. To determine if the MINICAM can be incorporated for first entry monitoring of chemical storage igloos.

1.4 TEST SCOPE. Testing of two MINICAMS will be conducted as follows:

a. Acceptance Test. The acceptance test will consist of subjecting detectors to specific concentrations of agent.

b. Field Functioning Test. A total of 64 trials will be conducted during the field functioning test, where each trial will consist of driving 30 minutes (MINICAM turned on for entire trial) and then stopping at a chemical storage igloo to sample.

1.5 TEST EQUIPMENT AND MATERIALS.

a. Two MINICAMS.

b. One "laboratory" type vehicle.

c. Two 75 foot heated tubes.

d. GB, GD, HD and VX agents as required.

e. Supply gas necessary to maintain operation of the MININCAM.

1.6 RE"PONSIBILITIES.

a. Test Engineering Branch, Test and Evaluation Office, CRDEC, will prepare test plan and test report.

b. Detection Directorate, CRDEC, will be responsible for providing MINCAM first entry monitoring system, for configuring power module and for installing system into van (vehicle supplied by Pine Bluff Aresenal).

c. Research, Development & Engineering Support Directorate, CRDEC will be responsible for overall test coordination.

d. Pine Bluff Arsenal will be responsible for test conduct and data reduction of agent sampling tests.

1.7 SAFETY. Agent testing will be conducted according to the Standard Operrating Procedure located in Appendix A. Testing will be conducted in accordance with safety directives and practices applicable to this test. Throughout testing, ar`` safety or health hazards encountered will be documented.

SECTION 2. DETAILED TEST PROCEDURES

2.1 ACCEPTANCE TEST

2.1.1 Objective

To validate the ability of the MINICAM to detect a range of time weighted average (TWA) concentration levels of GB, GD, HD and VX. The TWA is the sum or the products of the toxicant concentration and exposure duration divided by the total exposure time.

2.1.2 Criteria

For the MINICAMs to perform satisfactorily, 95% of the 1.0 ul TWA equivalent values (19 out of 20) must be in the range of 0.75 to 1.25 TWA. In addition 75% of the remaining values must be within + or - 25% of their theoretical values, except for the 0.2 TWA equivalent values where the results are desired but not required.

2.1.3 Required Data

Data will be recorded as shown on data sheet in Appendix B.

2.1.4 Data Acquistion Procedure

Testing will be conducted according to the standard operating procedure located in Appendix A and will be conducted in a laboratory with approved laboratory personnel. Two MINICAM's will be tested with GB, GD, HD and VX agents. Two separate series of injections will be performed with each series conducted as follows:

a. One blank (solvent) injection.

b. One 0.2 ul TWA equivalent injection.

c. Five 0.5 ul TWA equivalent injections.

d. Ten 1.0 ul TWA equivalent injections.

e. Five 2.0 ul TWA equivalent injections.

2.1.5 Analytical Procedure

After acceptance challenges have been performed, data collected will be analyzed. If the MINCAM's performed at an acceptable level then the field functional test can start. If the detectors do not perform at acceptable levels, the test will be put on hold until the necessary modifications and/or adjustments to the detectors have been made and retested until acceptance has been agreed upon.

2.2 FIELD FUNCTIONAL TEST

2.2.1 Objective.

To determine the ability of the MINICAM to perform first entry monitoring of a chemical munition storage igloo when mounted in a vehicle.

2.2.2 Criteria

The MINICAM first entry monitoring system shall provide a gain of accuracy over current procedues.

2.2.3 Required Data

Data will be recorded as shown on data sheets in Appendix B.

2.2.4 Data Acquisition Procedure

a. In preparation for the field test, a vehicle will be configured to provide the necessary power, support gases and data recording equipment needed to support two MINICAM's. All equipment will be checked for proper operation prior to test start.

b. Each day, prior to test start, the MINICAM's will be calibrated (only if necessary) according to the MINICAMS Operation and Maintenance Manual.

c. All agent testing will be conducted according to the SOP in Appendix A. Injections of agents shall be within +/- 25% of the TWA.

d. Testing will be conducted as follows:

(1) Turn on MINICAM's.

(2) Calibrate (first trial of day only) with GB.

(3) Drive 30 minutes on paved and secondary roads.

(4) Stop outside chemical munition storage igloo (empty).

(5) Connect 75 foot heated tube from each MINICAM through a vent hole in the igloo.

(6) Once inside the igloo, test personnel will use a syringe to inject VX TWA concentrations of 0.5 TWA, 1.0 TWA and 1.5 TWA into each heated tube. Agent testing will be conducted according to the SOP in Appendix A.

(7) If the MINICAM produces no reading following injections into the tube, the MINICAM will be directly injected with VX concentrations of 0.5 uL, 1.0 uL and 2.0 uL TWA to determine if MINICAM is functioning properly.

(8) Once a trial is completed, the tube will be removed from the igloo and and steps ~~1~~ 3 through 6 repeated until 64 trials are completed.

e. Preventive maintenance described in the MINICAMS Operation and Maintenance Manual will be performed as required.

f. Unscheduled maintenance performed will be documented as detailed in the Appendix B data sheet.

2.2.5 Analytical Procedure

Data will be compared to existing data of current first entry systems to determine if new MINICAM system is an improvement.

Prepared by:

JOHN JAGDE Test & Eval Ofc	DENNIS BOLT Detection Directorate

Recommending Approval:

MICHAEL SMITH
Chief, Test Eng Br, Test & Eval Ofc

Approved by:

ANTHONY J. SAPONARO
Chief, Test & Eval Ofc